Energy, Mines and Resources Canada | Énergie, Mines et Ressources Canada

CANMET

Canada Centre for Mineral and Energy Technology | Centre canadien de la technologie des minéraux et de l'énergie

PROPOSED DUST SAMPLING SYSTEM FOR MINES BASED ON A PORTABLE X-RAY DIFFRACTOMETER - A DISCUSSION PAPER

G. KNIGHT

ELLIOT LAKE LABORATORY

JUNE 1983

This document is an unedited interim report prepared primarily for discussion and internal reporting purposes. It does not represent a final opinion of the Canada Centre for Mineral and Energy Technology (CANMET)

Ce document est un rapport provisoire non-révisé et rédigé principalement pour lis de documentation interne. Il ne représente pas nécessairement l'opinion définitive du Centre canadien de la technologie des minéraux et de l'énergie (CANMET)

12 pp

MINERALS RESEARCH PROGRAM
MINING RESEARCH LABORATORIES
DIVISION REPORT MRP/MRL 83-54 (TR)

MICROMEDIA

PROPOSED DUST SAMPLING SYSTEM FOR MINES BASED ON A PORTABLE X-RAY DIFFRACTOMETER - A DISCUSSION PAPER

by

G. Knight*

ABSTRACT

A low cost X-ray diffractometer suitable for quartz assessment at mines and inspectors offices has been developed to the prototype stage. This paper discusses the elements required to produce a complete sampling and assessment system including the essential calibration procedures. This report is intended as the basis for a cooperative project between the Swedish manufacturer, U.S. mining agencies and CANMET.

---

* Research Scientist, Elliot Lake Laboratory, CANMET, Energy, Mines and Resources Canada, Elliot Lake, Ontario.

PROPOSITION D'UN SYSTÈME D'ÉCHANTILLONNAGE DE POUSSIÈRE DE MINES UTILISANT UN DIFFRACTOMÈTRE À RAYON X - DOCUMENT DE TRAVAIL

par

G. Knight*

RÉSUMÉ

On a mis au point le prototype d'un diffractomètre à rayon X peu coûteux qui convient à l'évaluation du quartz dans les mines et aux bureaux d'inspection. Ce document discute des éléments nécessaires à la conception d'un système complet d'échantillonnage et d'évaluation et inclue les étapes essentielles de calibration. Ce rapport constitue la base d'un projet de coopération entre les fabricants suédois, les agences américaines et le CANMET.

---

* Chercheur scientifique, Laboratoire d'Elliot Lake, CANMET, Énergie, Mines et Ressources Canada, Elliot Lake, Ontario.

INTRODUCTION

Airborne quartz dust is still considered a major health hazard in dusty industries including mining and requires continual efforts at control. At present, the trend in determination of quartz dust in mine air is to collect airborne dust on a filter using a small battery powered sampler and ship the filter to a central laboratory where it can be analyzed for quartz using an expensive diffractometer which may under computer control be capable of analyzing some 10,000 samples a year.

This system has the problem of a slow return of results dictated mainly by the shipping of the filters preventing the rapid feedback required in investigation of transient problems and delays in investigation and control of others.

The type of low cost unit as exemplified in the prototype developed by Mr. J. Baecklund of Metall AB Refina of Sweden could serve as the core on which a dust assessment system could be based at a mine giving the possibility of overnight results using the quality of technicians employed in mine ventilation departments of the larger mines with a hundred or more employees.

Such a system would have to address the significant problems in maintaining calibration without the dedicated personnel available in analytical laboratories.

This discussion paper outlines the objectives and components of such a complete system.

OBJECTIVES

1. Ease of use. Usable by personnel not trained in analytical techniques and control at the mine or local government inspector's office.
2. Reproducibility, sensitivity and accuracy. Studies in hard rock mines

have shown that full shift personal gravimetric samplers follow a logarithmic normal distribution with, for most individuals, a geometric standard deviation of 1.6 (1). The assessment system should be considerably more reproducible than this. A sensitivity of about 1/10 of the appropriate control limit is required. The control values used or foreseen in mining fall in the following ranges:

Respirable dust by mass 2 to 5 mg/m^3

Respirable combustible dust by mass 0.5 to 3 mg/m^3

Respirable quartz dust by 0.05 to 0.2 mg/m^3.

The accuracy of the system in assessing total annual exposure is expected to be within ±50%, however, this includes all the sampling errors and the system needs to be better.

Previous experience in hard rock mines using competent trained technicians and samplers collecting the dust from a volume of nearly 1 cubic metre in an eight hour shift suggests that for respirable dust by weight that: 95% of duplicate samples fall within the range of ±0.1 mg ± 10%, and that for respirable quartz, 90% falls within the range ± 0.01 mg ± 10%. Note: in the semi-trained (in gravimetric sampling techniques) technicians used at mines have higher error rates up to 30%.

The 5% of poor comparisons in respirable dust by mass appear to be due to mishandling and other human errors. The extra 5% in quartz assessment appears to be due to interference problems. It appears that a reasonable accuracy for the analytical system is 95% of samples within 25% of true value and that 95% of the means of seven samples should be within 10% of the true mean value with a sensitivity between 100 and 500 µg of quartz per cubic metre of air.

3. Calibration. The system should include facilities and techniques as required to maintain calibration of the following with a suggested schedule:

	Daily	Quarterly or Longer
Sampler air flow	X	
Sampler size selection		X
Sample weighing	X*	X
Sample ashing	?	X
Quartz analysis	X*	X

* carried out using standard samples which should be calibrated quarterly.

DUST SAMPLING

Respirable dust sampling is essential to assess the long term potential health hazard of pneumoconiosis as this is the major concern in dusty industries.

Two size selector specifications (SSP) are widely acceptable for assessment of workplace respirable dust (2,3). Fortunately most size selectors can give a reasonable approximation to either of these in the polydisperse mineral clouds present in mines by a suitable choice of the flow rate (4). With cyclones and impactors the mass collected on the filter is almost the same for both SSP's and thus the assessment method would be applicable to the same sampler used in either mode.

A running time indicator is useful in case of accidental or deliberate switching off in personal sampling.

FILTER

The size, type and porosity of the filter needs to be a compromise between sampling and assessment requirements. The present samplers produce 3 to 15 µg of respirable dust per sq mm and 70 to 600 ng of respirable quartz per sq mm in a full shift sample at the appropriate control concentration values. While values of dust thickness per unit area higher than this could be used at the control level, pump suction overload would occur more often

on the statistical distribution of high concentrations found in most mines and a target level of 6 μg per sq mm for eight hours sampling at 2 mg/m^3 respirable dust seems appropriate, that is, an airflow of 0.1 $ml/sec/mm^2$.

The proposed X-ray diffractometer requires an 'X-ray transparent' filter, i.e., thin and composed of low atomic weight elements. This also seems to be a suitable criterion for weight assessment by beta-ray attenuation and ash-combustible assessment by X-ray attenuation.

Filter mounting - filter mishandling by the technician is the major cause of damaged samples, and is likely to increase if thin filters are used. There would be substantial advantages in permanently attaching the filter to a rigid washer which would provide substantial mechanical protection to the dust deposit and the filter as well as closely defining the collection and assessment areas - particularly useful for the weight determinations by attenuation.

ASSESSMENT

X-ray diffraction assessment for quartz is the most critical requirement. Minimum area of filter to be examined - It has been determined that the maximum density is 150 ng per sq mm at the lowest control level of 0.05 mg/m^3 and that the sensitivity should be one tenth of this, that is 15 ng/mm^2. Considering the distribution of particle orientations and the small fraction that would be aligned a minimum of 200 particles is required. Two hundred particles, 3 μm in size and of density 2.6, weigh 14 ng, thus a minimum area of 1 mm^2 is essential and preferably it should be substantially greater. The suggested area for adequate resolution in the diffractometer is smaller than this and thus the sample should be moved so that the diffractometer traces a representative path over the surface of the filter to determine the average surface density.

Multiline analysis - The Elliot Lake Laboratory has found that significant interference occurs at about one third of the mines, and that in those mines it affects about one third of the samples. The Elliot Lake Laboratory uses multiline analysis with computer control to overcome this problem (5,6). In this technique the sample is always analyzed on two diffraction lines of quartz. If these do not agree within statistical limits the analysis proceeds to a third and if necessary a fourth line. The lines are chosen in any order from the four with Miller indices 1,0,0; 1,0,1; 1,1,2 and 2,1,1.

The computer control also selects counting time from two choices, determines some interference, i.e., peak offset from true position and rejects adjacent close peaks in background.

Beta-ray attenuation - could be used to determine the weight of respirable dust with greater accuracy and less technician training if it is built into the diffractometer, and its accuracy would depend only on the dust density on the filter and be independent of the area.

X-ray attenuation - could be used to determine the ash-combustible dust ratio fairly simply. However, calibration would probably vary from mine to mine depending on the proportion of heavy elements in the airborne mineral dust.

CALIBRATION PROCEDURES

RESPIRABLE SIZE SELECTION

The American Conference of Government Industrial Hygienist (ACGIH) recommends calibration using monodisperse dusts. This is complex and time consuming and I have not found publications on actual sampler configurations. The classic calibrations giving 1.7 L/min for the nylon cyclone included rarely used metal filter holders (7,8,9).

The Mine Safety and Health Agency (MSHA) has carried out calibrations collecting polydisperse coal dust and sizing by volume in a Coulter counter.

This method has difficulties in accurate transformation to aerodynamic size because of shape factors and particulate density.

The Elliot Lake Laboratory has followed the reference sampler method in which the physically definable horizontal elutriator is used as a reference size selector either in accordance with the British Medical Research Council specification or at 2/3 of that flow rate as a best match to the ACGIH specification or at 2/3 of that flow rate as a best match to the ACGIH specification in most polydisperse mineral dusts (4). Laboratory tests have shown that all size selectors can give reasonable agreement, by weight of respirable dust, with both horizontal elutriators to specifications within $\pm$ 10% in the wide range of mineral dust clouds produced in Elliot Lake Laboratory by suitable choice of flow rate. Theory suggests that substantial disagreement would only be expected in dust clouds with size distributions approaching monodisperse and well outside the normal polydisperse industrial range.

The horizontal elutriator reference dust sampler can be used with high reproducibility in the field if an electronically flow controlled sampling pump is used. The Elliot Lake Laboratory uses a Casella 113A horizontal elutriator and filter holder with a Dupont P2500 pump in the field with success. The high flow, 50 L/min, Casella Hexhlett sampler is easier to use in the laboratory because of smaller proportionate weighing errors.

FLOW CALIBRATION

The bubble tube is an absolute technique for determining flow rates to which secondary methods such as the taper tube flowmeter should be referenced. The high accuracy glass taper tube flowmeters such as the 1.7 to 2.1 L/min with a 3 cm scale manufactured by Rotameter Ltd. and marketed by Casella have been found useful for daily checking and are preferable to the plastic normal accuracy taper tube flowmeters often used, both because of readability and erosion changing calibration.

WEIGHTING CALIBRATION

The procedure to be adopted will depend on whether a balance or β-ray attenuation is used. Details are not proposed at this time, but will have to be set up for the system chosen.

X-RAY ATTENUATION CALIBRATION

The ash-combustible dust calibration will depend on the specific mineral composition at the mine, particularly the heavier elements, however, it is probably possible to set up a program to determine the calibration using the samples collected in a hard rock mine by looking for the extremes of the X-ray/β-ray attenuation ratios. Those with a high ratio would be almost pure mineral, as from the crushing area, those with a low ratio, almost pure diesel exhaust. Alternatively, these could be determined for each mine by examining samples at a central laboratory by normal ashing techniques.

X-RAY DIFFRACTION CALIBRATION

No sampler has yet been found to give a precisely uniform dust deposition; most give a symmetric radial distribution and calibration should be determined by collection of samples by the chosen sampler in a pure quartz airborne dust cloud (6).

This primary calibration could be carried out at central laboratory and secondary standards be prepared and calibrated for shipping to individual mines with X-ray diffractometers.

A small proportion of samples should be mailed on to the central laboratory as a quality control, and could be used to detect drifts in the secondary standards and possibly determine the adjustment or call for recalibration by the central laboratory. This check by the central laboratory could also be used to determine more carefully the quartz present when interference

occurs and recommend a specific analytical program, i.e., a diffraction line order other than the one 1,0,1; 1,1,2; 2,1,1 and 1,0,0 found efficient for most mines.

DISCUSSION

The proposal is put forward that a sampling and assessment system could be developed for mines using small low cost X-ray diffractometers if it is backed up by a central laboratory with trained analysts to supply the essential calibration facilities. The Metall AB Refina prototype X-ray diffractometer looks very promising.

The addition of β-ray and X-ray attenuation could decrease the technician input at the mine and render the design of the dust sampler more flexible in that a smaller filter, smaller sampling head, lower airflow and smaller pumps could be used than the existing generation of personal gravimetric samplers.

The use of a rigid washer attached to a thin probably fragile filter should decrease the proportion of filters lost in handling by the technicians both while learning and when experienced. Turnover rates in the mines seen to be about once per year for these technicians and learning delicate handling occupies a few months.

REFERENCES

1. Knight, G., Kirk, B, Sutarno, R. and Stefanich, W., "Personal dust sampling in mines: statistical analysis"; Proc. 2nd Int. Mine Ventilation Congress, Reno, Nevada, November 4-8, 1979 (Ed. P. Mousset-Jones); AIME, New York 1980; p 303.

2. Anon. Medical Research Council; in Inhaled Particles and Vapours; (Ed.) C.N. Davies; Pergamon Press, Oxford, 1961; p 475.

3. Anon., "Threshold limit values of airborne contaminants"; Am Conf of Govt Ind. Hyg, Cincinnatti, 1974.

4. Knight, G. and Kirk, B., "Comparison of respirable dust specification with recent lung data"; Am Ind Hyg Assoc J, 43, pp 575-579; Aug 1982.

5. Knight, G., "Mine dust sampling system - CAMPEDS"; CANMET Report78-7; CANMET, Energy, Mines and Resources Canada; 1978.

6. Knight, G., "The use of an on line computer to control quantitative XRD analysis"; Proc. 4th Symp. on Electron Microscopy and X-Ray Application to Engineering and Occup. Health Analysis, State College, Penn; Oct 1980.

7. Knuth, R.H., "Recalibration of size selective samplers"; Am Ind Hyg Assoc J, vol 30 p 379; 1969.

8. Ettinger, M.J., Jennings, E. and Royer, G.W., "Calibration of two stage air samplers"; Am Ind Hyg Assoc J, vol 31, p 5337; 1970.

9. Sutton, unpublished report.